

DOCKET NO: 278600US0PCT

IN THE UNITED STATES PATENT & TRADEMARK OFFICE

IN RE APPLICATION OF :
ULRIKE LICHT, ET AL. : EXAMINER: NILAND, P.
SERIAL NO: 10/553,037 :
FILED: OCTOBER 11, 2005 : GROUP ART UNIT: 1796
FOR: SELF-EMULSIFYING AQUEOUS :
POLYURETHANE DISPERSIONS

APPEAL BRIEF

COMMISSIONER FOR PATENTS
ALEXANDRIA, VIRGINIA 22313

SIR:

This is an appeal from the Examiner's Final Rejection dated January 23, 2009, of Claims 8, 9, 21-38 and 40-56. A Notice of Appeal was filed on April 23, 2009.

I. REAL PARTY IN INTEREST

The real party in interest is BASF SE of Ludwigshafen, Germany, by virtue of the assignment recorded June 22, 2009, at Reel/Frame 022853/0040.

II. RELATED APPEALS AND INTERFERENCES

Appellants, Appellants' legal representative and their assignee are not aware of any other appeals or interferences which will directly affect or be directly affected by or having a bearing on the Board's decision in this appeal.

III. STATUS OF CLAIMS

The appealed claims are Claims 8, 9, 21-38 and 40-56. Claims 8, 9, 21-38 and 40-56 stand rejected.

The status of Claims 8, 9, 21-38 and 40-56 is “previously presented”. The status of Claims 1-7, 10-20 and 39 is “canceled”.

IV. STATUS OF AMENDMENTS

No Amendment under 37 C.F.R. §1.116 was filed.

V. SUMMARY OF CLAIMED SUBJECT MATTER

As claimed in **Claim 8**, the present application relates to process for preparing a primary dispersion, said process comprising:

reacting the following components a), b1), and c) and optionally b2), optionally b3), and optionally b4) in the presence of water, thereby obtaining an aqueous primary dispersion, which comprises at least one polyurethane;

wherein

a) is at least one polyisocyanate,

b1) is at least one polyol comprising a structural unit $[-CH_2-CH_2-O-]_w$, wherein w is a positive integer from 10 to 200, wherein said structural unit $[-CH_2-CH_2-O-]_w$ is obtained from a synthesis component selected from the group consisting of ethylene glycol, polyethylene glycol having a number average molecular weight of between 106 and 2000, and ethylene oxide;

b2) is at least one polyol other than b1),

b3) is at least one compound containing at least two isocyanate-reactive groups selected from the group consisting of thiol groups and primary and secondary amino groups,

b4) is at least one monofunctional monomer having an isocyanate-reactive group, and
c) is at least one ionic or potentially ionic synthesis component,
wherein the component c) is represented by the general formula $RG-R^1-DG$,
wherein RG is at least one isocyanate reactive group,
DG is at least one actively dispersing group, and
 R^1 is an aliphatic, cycloaliphatic or aromatic radical comprising 1 to 20 carbon atoms;
wherein
the fraction of the structural units $-[-CH_2-CH_2-O]-$, calculated at 44 g/mol, in the
polyol b1) is from 10 to 90% by weight, and
the fraction of the structural units $-[-CH_2-CH_2-O]-$, calculated at 44 g/mol, in the
sum of the components a) + b1) + b2) + b3) + b4) + c) is at least 3% by weight.

See for example, page 2, lines 10-35, and page 9, lines 14-26, and the Examples of the
specification.

Claim 27 relates to a process for preparing a primary dispersion, said process
comprising:

reacting the following components a), b1), and optionally b2), optionally b3),
optionally b4) and optionally c) in the presence of water, thereby obtaining an aqueous primary
dispersion, which comprises at least one polyurethane;

wherein

first all components are mixed with water, to obtain an emulsion having a water
phase,

then said emulsion is heated,

after the reaction temperature has been reached, a catalyst is added to the water phase of said emulsion, and

wherein

a) is at least one polyisocyanate,

b1) is at least one polyol comprising a structural unit $[-CH_2-CH_2-O-]_w$, wherein w is a positive integer from 10 to 200, wherein said structural unit $[-CH_2-CH_2-O-]_w$ is obtained from a synthesis component selected from the group consisting of ethylene glycol, polyethylene glycol having a number average molecular weight of between 106 and 2000, and ethylene oxide;

b2) is at least one polyol other than b1),

b3) is at least one compound containing at least two isocyanate-reactive groups selected from the group consisting of thiol groups and primary and secondary amino groups,

b4) is at least one monofunctional monomer having an isocyanate-reactive group, and

c) is at least one ionic or potentially ionic synthesis component,

wherein the component c) is represented by the general formula $RG-R^1-DG$,

wherein RG is at least one isocyanate reactive group,

DG is at least one actively dispersing group, and

R^1 is an aliphatic, cycloaliphatic or aromatic radical comprising 1 to 20 carbon atoms;

wherein

the fraction of the structural units $[-CH_2-CH_2-O-]$, calculated at 44 g/mol, in the polyol b1) is from 10 to 90% by weight, and

the fraction of the structural units $[-CH_2-CH_2-O-]$, calculated at 44 g/mol, in the sum of the components a) + b1) + b2) + b3) + b4) + c) is at least 3% by weight.

See for example, page 2, lines 10-35, and page 9, lines 14-26, page 12, lines 9-19 and

the Examples of the specification.

Claim 54 relates to a process for preparing a primary dispersion, said process comprising:

reacting the following components a), b1), and optionally b2), optionally b3), optionally b4) and optionally c) in the presence of water, thereby obtaining an aqueous primary dispersion, which comprises at least one polyurethane;

wherein

first all components are mixed with water, to obtain an emulsion having a water phase and an oil phase,

then said emulsion is heated,

after the reaction temperature has been reached, a catalyst is added to the oil phase of said emulsion, and

wherein

a) is at least one polyisocyanate,

b1) is at least one polyol comprising a structural unit $[-CH_2-CH_2-O-]_w$, wherein w is a positive integer from 10 to 200, wherein said structural unit $[-CH_2-CH_2-O-]_w$ is obtained from a synthesis component selected from the group consisting of ethylene glycol, polyethylene glycol having a number average molecular weight of between 106 and 2000, and ethylene oxide;

b2) is at least one polyol other than b1),

b3) is at least one compound containing at least two isocyanate-reactive groups selected from the group consisting of thiol groups and primary and secondary amino groups,

b4) is at least one monofunctional monomer having an isocyanate-reactive group, and

c) is at least one ionic or potentially ionic synthesis component,

wherein the component c) is represented by the general formula $RG-R^1-DG$,
wherein RG is at least one isocyanate reactive group,
DG is at least one actively dispersing group, and
 R^1 is an aliphatic, cycloaliphatic or aromatic radical comprising 1 to 20 carbon atoms;
wherein
the fraction of the structural units $[-CH_2-CH_2-O-]$, calculated at 44 g/mol, in the
polyol b1) is from 10 to 90% by weight, and
the fraction of the structural units $[-CH_2-CH_2-O-]$, calculated at 44 g/mol, in the
sum of the components a) + b1) + b2) + b3) + b4) + c) is at least 3% by weight.

See for example, page 2, lines 10-35, and page 9, lines 14-26, page 12, lines 9-24 and
the Examples of the specification.

Claim 55 relates to a process for preparing a primary dispersion, said process
comprising:

reacting the following components a), b1), and optionally b2), optionally b3),
optionally b4) and optionally c) in the presence of water, thereby obtaining an aqueous primary
dispersion, which comprises at least one polyurethane;

wherein

first all components are mixed with water, to obtain an emulsion having a water
phase and an oil phase, a catalyst is added to the water phase of said emulsion,
then said emulsion is heated, and

wherein

a) is at least one polyisocyanate,

b1) is at least one polyol comprising a structural unit $[-CH_2-CH_2-O-]_w$, wherein w is a positive integer from 10 to 200, wherein said structural unit $[-CH_2-CH_2-O-]_w$ is obtained from a synthesis component selected from the group consisting of ethylene glycol, polyethylene glycol having a number average molecular weight of between 106 and 2000, and ethylene oxide;

b2) is at least one polyol other than b1),

b3) is at least one compound containing at least two isocyanate-reactive groups selected from the group consisting of thiol groups and primary and secondary amino groups,

b4) is at least one monofunctional monomer having an isocyanate-reactive group, and

c) is at least one ionic or potentially ionic synthesis component,

wherein the component c) is represented by the general formula $RG-R^1-DG$,

wherein RG is at least one isocyanate reactive group,

DG is at least one actively dispersing group, and

R^1 is an aliphatic, cycloaliphatic or aromatic radical comprising 1 to 20 carbon atoms;

wherein

the fraction of the structural units $[-CH_2-CH_2-O-]$, calculated at 44 g/mol, in the polyol b1) is from 10 to 90% by weight, and

the fraction of the structural units $[-CH_2-CH_2-O-]$, calculated at 44 g/mol, in the sum of the components a) + b1) + b2) + b3) + b4) + c) is at least 3% by weight.

See for example, page 2, lines 10-35, and page 9, lines 10-26, page 12, lines 9-24 and the Examples of the specification.

VI. GROUNDS OF REJECTION TO BE REVIEWED ON APPEAL

(A) Claims 8, 9, 21-38 and 40-56 stand rejected as under 35 U.S.C. § 112, 1st paragraph as failing to comply with the written description requirement.

(B) Claims 8, 9, 21-38 and 40-56 stand rejected as being obvious under 35 U.S.C. §103 (a) over Licht et al (WO 02/064657 as translated by US 2004/0077777 A1) in view of Jakubowski et al (US 5,959,027) and Scriven et al (US 4,046,729).

VII. ARGUMENT

Ground (A)

Claims 8, 9, 21-38 and 40-56 stand rejected as under 35 U.S.C. § 112, 1st paragraph as failing to comply with the written description requirement. That rejection is untenable and should not be sustained.

The subject matter of the claims was described in the original specification in such a way as to reasonably convey to one skilled in the relevant art that the inventors were in possession in of the claimed invention at the time the application was filed.

The molecular weight of the polyethylene glycol is a number average as supported, for example at page 16, line 6, of the specification. DIN 53240 describes the determination of the OH number. A translation of DIN 53240 was filed on March 13, 2008. The OH-number is always unambiguously connected with exactly one (average) molecular weight of a sample, see topic #9 of the English translation of DIN 53240. The OH-number means how much potassium hydroxide is consumed on neutralization of an OH group containing sample and is usually given in mg KOH per g sample. Since 1 mol KOH has a molecular weight of 56.1 g/mol, an OH-number of 100 means a consumption of 100 mg KOH/g=1.8 mmol KOH/g. This means that 1 g of the sample contains 1.8 mmol OH-groups. Thus, for a diol (two OH functions) the average molecular weight of the sample is 1111 g/mol. In case the molecular

weight of the sample is distributed over a wide range, the OH number refers to average molecular weight.

Thus, the rejection of Claims 8, 9, 21-38 and 40-56 under 35 U.S.C. § 112, 1st paragraph, should be REVERSED.

Ground (B)

Claims 8, 9, 21-38 and 40-56 stand rejected as being obvious under 35 U.S.C. §103 (a) over Licht et al (WO 02/064657 as translated by US 2004/0077777 A1) in view of Jakubowski et al (US 5,959,027) and Scriven et al (US 4,046,729). That rejection is untenable and should not be sustained.

It is an object of the present invention to provide primary dispersions which comprise polyurethane, which are finely divided without the use of high shear forces, and which make it possible not only for the raw materials to be emulsified finely but also for the products to be dispersed. See page 2, 1st full paragraph of the specification.

However, WO 02/064657 (US equivalent 2004/0077777) in view of US 5,959,027 and US 4,046,729 fail to disclose or suggest the process of producing the aqueous primary dispersions as claimed in Claims 8, 27, 54 and 55.

According to US 2004/0077777, paragraph [0014] "incorporation of ionically or non-ionically hydrophilic groups" is **not necessary**. Thus, there is no motivation to combine with other documents disclosing emulsifiers for polyisocyanates in US 2004/0077777 since this reference teaches away from doing so.

Further, US 4046729 discloses "the reaction product of ethylene glycol with a mixture of propylene oxide and ethylene oxide" (col. 8, line 49) as well as salt groups which may be anionic or cationic (col. 11, lines 1 to 36). However, the polyalkylene ether polyol according to US 4046729 contains **2 to 6** alkylene oxide units (col. 8, line 46) while the polyols of the present invention contain more alkylene oxide units than US 4046729, namely **10 to 200**.

US 5,959027 is silent about mixed polyalkylene oxides.

Thus, even if WO 02/064657 (US equivalent 2004/0077777) and US 5,959027 and US 4046729 were combined, the present invention does not result.

Further regarding the specific structures of component c), Applicants wish to refer to dependent Claims 43, 44, 50 and 51.

“wherein the component c) is selected from the group consisting of monohydroxycarboxylic acids, monohydroxysulfonic acids, monoaminocarboxylic acids, monoaminosulfonic acids and mixtures thereof”

And

“wherein the component c) is selected from the group consisting of mercaptoacetic acid, mercaptopropionic acid, thiolactic acid, mercaptosuccinic acid, glycine, iminodiacetic acid, sarcosine, alanine, β -alanine, leucine, isoleucine, aminobutyric acid, hydroxyacetic acid, hydroxypivalic acid, lactic acid, hydroxysuccinic acid, hydroxydecanoic acid, dimethylolpropionic acid, dimethylolbutyric acid, ethylenediaminetriacetic acid, hydroxydodecanoic acid, hydroxyhexadecanoic acid, 12-hydroxystearic acid, aminonaphthalenecarboxylic acid, hydroxyethanesulfonic acid, hydroxypropanesulfonic acid, mercaptoethanesulfonic acid, mercaptopropanesulfonic acid, aminomethanesulfonic acid, taurine, aminopropanesulfonic acid and mixtures thereof.”

In Claim 54, the catalyst is added to the oil phase of the emulsion. The claim is otherwise analogous to Claim 27.

In Claim 55, the catalyst is added to the water phase of the emulsion followed by heating of the emulsion. The claim is otherwise analogous to Claim 27.

In Claim 56, which depends on Claim 27, component c) is not present.

Claims 54-56 are separately patentable because Licht et al (WO 02/064657 as translated by US 2004/0077777 A1), Jakubowski et al (US 5,959,027) and Scriven et al (US 4,046,729), alone or in combination, fail to disclose or suggest that the catalyst is added to the oil phase of the emulsion; that the catalyst is added to the water phase of the emulsion followed by heating of the emulsion; or that component c) is not present.

Claims 9 and 28:

Claims 9 and 28 are separately patentable because Licht et al (WO 02/064657 as translated by US 2004/0077777 A1), Jakubowski et al (US 5,959,027) and Scriven et al (US 4,046,729), alone or in combination, fail to disclose or suggest that dispersing takes place with shear forces below 10^8 W/cm³.

Claims 21 and 29:

Claims 21 and 29 are separately patentable because Licht et al (WO 02/064657 as translated by US 2004/0077777 A1), Jakubowski et al (US 5,959,027) and Scriven et al (US 4,046,729), alone or in combination, fail to disclose or suggest that the number average molecular weight of the polyol b1) is 500 to 2000 g/mol.

Claims 22 and 30:

Claims 22 and 30 are separately patentable because Licht et al (WO 02/064657 as translated by US 2004/0077777 A1), Jakubowski et al (US 5,959,027) and Scriven et al (US 4,046,729), alone or in combination, fail to disclose or suggest that the polyol b1) is a copolymer comprising ethylene oxide and propylene oxide.

Claims 23 and 31:

Claims 23 and 31 are separately patentable because Licht et al (WO 02/064657 as translated by US 2004/0077777 A1), Jakubowski et al (US 5,959,027) and Scriven et al (US 4,046,729), alone or in combination, fail to disclose or suggest that the copolymer is a block copolymer.

Claims 24 and 32:

Claims 24 and 32 are separately patentable because Licht et al (WO 02/064657 as translated by US 2004/0077777 A1), Jakubowski et al (US 5,959,027) and Scriven et al (US 4,046,729), alone or in combination, fail to disclose or suggest that the polyol b1) comprises at least one terminal structural unit -CH₂-O-H.

Claims 25 and 33:

Claims 25 and 33 are separately patentable because Licht et al (WO 02/064657 as translated by US 2004/0077777 A1), Jakubowski et al (US 5,959,027) and Scriven et al (US 4,046,729), alone or in combination, fail to disclose or suggest that the polyol b1) is a polyesterol.

Claims 26 and 34:

Claims 26 and 34 are separately patentable because Licht et al (WO 02/064657 as

translated by US 2004/0077777 A1), Jakubowski et al (US 5,959,027) and Scriven et al (US 4,046,729), alone or in combination, fail to disclose or suggest that the z-average particle size as measured by dynamic light scattering using the Malvern® Autosizer 2 C is below 100 nm.

Claim 35:

Claim 35 is separately patentable because Licht et al (WO 02/064657 as translated by US 2004/0077777 A1), Jakubowski et al (US 5,959,027) and Scriven et al (US 4,046,729), alone or in combination, fail to disclose or suggest a method of coating a substrate, comprising:
applying the aqueous primary dispersion obtained by the process of Claim 8 to the substrate, thereby coating the substrate.

Claims 36 and 38:

Claims 36 and 38 are separately patentable because Licht et al (WO 02/064657 as translated by US 2004/0077777 A1), Jakubowski et al (US 5,959,027) and Scriven et al (US 4,046,729), alone or in combination, fail to disclose or suggest that the substrate comprises a material selected from the group consisting of wood, wood veneer, paper, board, card, textile, leather, nonwoven, plastic, glass, ceramic, metals, coated metals, and mineral building materials.

Claim 37:

Claim 37 is separately patentable because Licht et al (WO 02/064657 as translated by US 2004/0077777 A1), Jakubowski et al (US 5,959,027) and Scriven et al (US 4,046,729), alone or in combination, fail to disclose or suggest a method of coating a substrate, comprising:

applying the aqueous primary dispersion obtained by the process of claim 27 to the substrate, thereby coating the substrate.

Claims 40 and 47:

Claims 40 and 47 are separately patentable because Licht et al (WO 02/064657 as translated by US 2004/0077777 A1), Jakubowski et al (US 5,959,027) and Scriven et al (US 4,046,729), alone or in combination, fail to disclose or suggest that RG is -OH.

Claims 41 and 48:

Claims 41 and 48 are separately patentable because Licht et al (WO 02/064657 as translated by US 2004/0077777 A1), Jakubowski et al (US 5,959,027) and Scriven et al (US 4,046,729), alone or in combination, fail to disclose or suggest that RG is NHR^2 , wherein R^2 is methyl, ethyl, isopropyl, n-propyl, n-butyl, iso-butyl, sec-butyl, tert-butyl, cyclopentyl or cyclohexyl.

Claims 42 and 49:

Claims 42 and 49 are separately patentable because Licht et al (WO 02/064657 as translated by US 2004/0077777 A1), Jakubowski et al (US 5,959,027) and Scriven et al (US 4,046,729), alone or in combination, fail to disclose or suggest that RG is NHR^2 , wherein R^2 is cyclohexyl.

Claims 43 and 50:

Claims 43 and 50 are separately patentable because Licht et al (WO 02/064657 as translated by US 2004/0077777 A1), Jakubowski et al (US 5,959,027) and Scriven et al (US 4,046,729), alone or in combination, fail to disclose or suggest that the component c) is selected from the group consisting of monohydroxycarboxylic acids, monohydroxysulfonic acids, monoaminocarboxylic acids, monoaminosulfonic acids and mixtures thereof.

Claims 44 and 51:

Claims 44 and 51 are separately patentable because Licht et al (WO 02/064657 as translated by US 2004/0077777 A1), Jakubowski et al (US 5,959,027) and Scriven et al (US 4,046,729), alone or in combination, fail to disclose or suggest that the component c) is selected from the group consisting of mercaptoacetic acid, mercaptopropionic acid, thiolactic acid, mercaptosuccinic acid, glycine, iminodiacetic acid, sarcosine, alanine, β -alanine, leucine, isoleucine, aminobutyric acid, hydroxyacetic acid, hydroxypivalic acid, lactic acid, hydroxysuccinic acid, hydroxydecanoic acid, dimethylolpropionic acid, dimethylolbutyric acid, ethylenediaminetriacetic acid, hydroxydodecanoic acid, hydroxyhexadecanoic acid, 12-hydroxystearic acid, aminonaphthalenecarboxylic acid, hydroxyethanesulfonic acid, hydroxypropanesulfonic acid, mercaptoethanesulfonic acid, mercaptopropanesulfonic acid, aminomethanesulfonic acid, taurine, aminopropanesulfonic acid and mixtures thereof.

Claims 45 and 46:

Claims 45 and 46 are separately patentable because Licht et al (WO 02/064657 as translated by US 2004/0077777 A1), Jakubowski et al (US 5,959,027) and Scriven et al (US 4,046,729), alone or in combination, fail to disclose or suggest that in a first step, an organic phase is prepared homogeneously and in a second step said organic phase is added to a water phase or a water phase is added to the organic phase.

Claims 52 and 53:

Claims 52 and 53 are separately patentable because Licht et al (WO 02/064657 as translated by US 2004/0077777 A1), Jakubowski et al (US 5,959,027) and Scriven et al (US 4,046,729), alone or in combination, fail to disclose or suggest that RG is -OH, -SH, -NH₂, or -NHR², where R² is methyl, ethyl, iso-propyl, n-propyl, n-butyl, isobutyl, sec-butyl, tert-butyl, cyclopentyl or cyclohexyl.

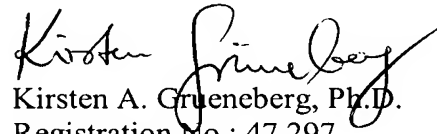
Thus, Claims 8, 9, 21-38 and 40-56 are Not obvious over Licht et al (WO 02/064657 as translated by US 2004/0077777 A1) in view of Jakubowski et al (US 5,959,027) and Scriven et al (US 4,046,729) within the meaning of 35 U.S.C. §103(a). For all the above reasons, it is respectfully requested that this rejection be REVERSED.

CONCLUSION

For the above reasons, it is respectfully requested that all the rejections still pending in the Final Office Action be REVERSED.

Respectfully submitted,

OBLON, SPIVAK, McCLELLAND,
MAIER & NEUSTADT, P.C.


Kirsten A. Grueneberg, Ph.D.
Registration No.: 47,297

VIII. CLAIMS APPENDIX

Claim 8: A process for preparing a primary dispersion, said process comprising:
reacting the following components a), b1), and c) and optionally b2), optionally b3),
and optionally b4) in the presence of water, thereby obtaining an aqueous primary dispersion,
which comprises at least one polyurethane;

wherein

a) is at least one polyisocyanate,

b1) is at least one polyol comprising a structural unit $[-CH_2-CH_2-O-]_w-$, wherein w
is a positive integer from 10 to 200, wherein said structural unit $[-CH_2-CH_2-O-]_w-$ is obtained
from a synthesis component selected from the group consisting of ethylene glycol,
polyethylene glycol having a number average molecular weight of between 106 and 2000, and
ethylene oxide;

b2) is at least one polyol other than b1),

b3) is at least one compound containing at least two isocyanate-reactive groups
selected from the group consisting of thiol groups and primary and secondary amino groups,

b4) is at least one monofunctional monomer having an isocyanate-reactive group, and

c) is at least one ionic or potentially ionic synthesis component,

wherein the component c) is represented by the general formula $RG-R^1-DG$,

wherein RG is at least one isocyanate reactive group,

DG is at least one actively dispersing group, and

R^1 is an aliphatic, cycloaliphatic or aromatic radical comprising 1 to 20 carbon atoms;

wherein

the fraction of the structural units $[-CH_2-CH_2-O-]$, calculated at 44 g/mol, in the
polyol b1) is from 10 to 90% by weight, and

the fraction of the structural units $[-CH_2-CH_2-O-]$, calculated at 44 g/mol, in the sum of the components a) + b1) + b2) + b3) + b4) + c) is at least 3% by weight.

Claim 9: The process according to claim 8, wherein dispersing takes place with shear forces below 10^8 W/cm³.

Claim 21: The process according to claim 8, wherein the number average molecular weight of the polyol b1) is 500 to 2000 g/mol.

Claim 22: The process according to claim 8, wherein the polyol b1) is a copolymer comprising ethylene oxide and propylene oxide.

Claim 23: The process according to claim 22, wherein the copolymer is a block copolymer.

Claim 24: The process according to claim 8, wherein the polyol b1) comprises at least one terminal structural unit $-CH_2-O-H$.

Claim 25: The process according to claim 8, wherein the polyol b1) is a polyesterol.

Claim 26: The process according to claim 8, wherein the z-average particle size as measured by dynamic light scattering using the Malvern® Autosizer 2 C is below 100 nm.

Claim 27: A process for preparing a primary dispersion, said process comprising: reacting the following components a), b1), and optionally b2), optionally b3),

optionally b4) and optionally c) in the presence of water, thereby obtaining an aqueous primary dispersion, which comprises at least one polyurethane;

wherein

first all components are mixed with water, to obtain an emulsion having a water phase,

then said emulsion is heated,

after the reaction temperature has been reached, a catalyst is added to the water phase of said emulsion, and

wherein

a) is at least one polyisocyanate,

b1) is at least one polyol comprising a structural unit $[-CH_2-CH_2-O-]_w-$, wherein w is a positive integer from 10 to 200, wherein said structural unit $[-CH_2-CH_2-O-]_w-$ is obtained from a synthesis component selected from the group consisting of ethylene glycol, polyethylene glycol having a number average molecular weight of between 106 and 2000, and ethylene oxide;

b2) is at least one polyol other than b1),

b3) is at least one compound containing at least two isocyanate-reactive groups selected from the group consisting of thiol groups and primary and secondary amino groups,

b4) is at least one monofunctional monomer having an isocyanate-reactive group, and

c) is at least one ionic or potentially ionic synthesis component,

wherein the component c) is represented by the general formula $RG-R^1-DG$,

wherein RG is at least one isocyanate reactive group,

DG is at least one actively dispersing group, and

R^1 is an aliphatic, cycloaliphatic or aromatic radical comprising 1 to 20 carbon atoms;

wherein

the fraction of the structural units $[-CH_2-CH_2-O-]$, calculated at 44 g/mol, in the polyol b1) is from 10 to 90% by weight, and

the fraction of the structural units $[-CH_2-CH_2-O-]$, calculated at 44 g/mol, in the sum of the components a) + b1) + b2) + b3) + b4) + c) is at least 3% by weight.

Claim 28: The process according to claim 27, wherein dispersing takes place with shear forces below 10^8 W/cm³.

Claim 29: The process according to claim 27, wherein the number average molecular weight of the polyol b1) is 500 to 2000 g/mol.

Claim 30: The process according to claim 27, wherein the polyol b1) is a copolymer comprising ethylene oxide and propylene oxide.

Claim 31: The process according to claim 30, wherein the copolymer is a block copolymer.

Claim 32: The process according to claim 27, wherein the polyol b1) comprises at least one terminal structural unit $-CH_2-O-H$.

Claim 33: The process according to claim 27, wherein the polyol b1) is a polyesterol.

Claim 34: The process according to claim 27, wherein the z-average particle size as measured by dynamic light scattering using the Malvern® Autosizer 2 C is below 100 nm.

Claim 35: A method of coating a substrate, comprising:
applying the aqueous primary dispersion obtained by the process of Claim 8 to the substrate, thereby coating the substrate.

Claim 36: The method of claim 35, wherein the substrate comprises a material selected from the group consisting of wood, wood veneer, paper, board, card, textile, leather, nonwoven, plastic, glass, ceramic, metals, coated metals, and mineral building materials.

Claim 37: A method of coating a substrate, comprising:
applying the aqueous primary dispersion obtained by the process of claim 27 to the substrate, thereby coating the substrate.

Claim 38: The method of claim 37, wherein the substrate comprises a material selected from the group consisting of wood, wood veneer, paper, board, card, textile, leather, nonwoven, plastic, glass, ceramic, metals, coated metals, and mineral building materials.

Claim 40: The process according to claim 8, wherein RG is -OH.

Claim 41: The process according to claim 8, wherein RG is NHR^2 , wherein R^2 is methyl, ethyl, isopropyl, n-propyl, n-butyl, iso-butyl, sec-butyl, tert-butyl, cyclopentyl or cyclohexyl.

Claim 42: The process according to claim 8, wherein RG is NHR^2 , wherein R^2 is cyclohexyl.

Claim 43: The process according to claim 8, wherein the component c) is selected from the group consisting of monohydroxycarboxylic acids, monohydroxysulfonic acids, monoaminocarboxylic acids, monoaminosulfonic acids and mixtures thereof.

Claim 44: The process according to claim 8, wherein the component c) is selected from the group consisting of mercaptoacetic acid, mercaptopropionic acid, thiolactic acid, mercaptosuccinic acid, glycine, iminodiacetic acid, sarcosine, alanine, β -alanine, leucine, isoleucine, aminobutyric acid, hydroxyacetic acid, hydroxypivalic acid, lactic acid, hydroxysuccinic acid, hydroxydecanoic acid, dimethylolpropionic acid, dimethylolbutyric acid, ethylenediaminetriacetic acid, hydroxydodecanoic acid, hydroxyhexadecanoic acid, 12-hydroxystearic acid, aminonaphthalenecarboxylic acid, hydroxyethanesulfonic acid, hydroxypropanesulfonic acid, mercaptoethanesulfonic acid, mercaptopropanesulfonic acid, aminomethanesulfonic acid, taurine, aminopropanesulfonic acid and mixtures thereof.

Claim 45: The process according to claim 8, wherein in a first step, an organic phase is prepared homogeneously and in a second step said organic phase is added to a water phase or a water phase is added to the organic phase.

Claim 46: The process according to claim 27, wherein in a first step, an organic phase is prepared homogeneously and in a second step said organic phase is added to a water phase or a water phase is added to the organic phase.

Claim 47: The process according to claim 27, wherein RG is -OH.

Claim 48: The process according to claim 27, wherein RG is NHR^2 , wherein R^2 is

methyl, ethyl, isopropyl, n-propyl, n-butyl, iso-butyl, sec-butyl, tert-butyl, cyclopentyl or cyclohexyl.

Claim 49: The process according to claim 27, wherein RG is NHR^2 , wherein R^2 is cyclohexyl.

Claim 50: The process according to claim 27, wherein the component c) is selected from the group consisting of monohydroxycarboxylic acids, monohydroxysulfonic acids, monoaminocarboxylic acids, monoaminosulfonic acids and mixtures thereof.

Claim 51: The process according to claim 27, wherein the component c) is selected from the group consisting of mercaptoacetic acid, mercaptopropionic acid, thiolactic acid, mercaptosuccinic acid, glycine, iminodiacetic acid, sarcosine, alanine, β -alanine, leucine, isoleucine, aminobutyric acid, hydroxyacetic acid, hydroxypivalic acid, lactic acid, hydroxysuccinic acid, hydroxydecanoic acid, dimethylolpropionic acid, dimethylolbutyric acid, ethylenediaminetriacetic acid, hydroxydodecanoic acid, hydroxyhexadecanoic acid, 12-hydroxystearic acid, aminonaphthalenecarboxylic acid, hydroxyethanesulfonic acid, hydroxypropanesulfonic acid, mercaptoethanesulfonic acid, mercaptopropanesulfonic acid, aminomethanesulfonic acid, taurine, aminopropanesulfonic acid and mixtures thereof.

Claim 52: The process according to claim 8, wherein RG is -OH, -SH, -NH₂, or -NHR², where R^2 is methyl, ethyl, iso-propyl, n-propyl, n-butyl, isobutyl, sec-butyl, tert-butyl, cyclopentyl or cyclohexyl.

Claim 53: The process according to claim 27, wherein RG is -OH, -SH, -NH₂, or

-NHR², wherein R² is methyl, ethyl, iso-propyl, n-propyl, n-butyl, isobutyl, sec-butyl, tert-butyl, cyclopentyl or cyclohexyl.

Claim 54: A process for preparing a primary dispersion, said process comprising:
reacting the following components a), b1), and optionally b2), optionally b3),
optionally b4) and optionally c) in the presence of water, thereby obtaining an aqueous primary
dispersion, which comprises at least one polyurethane;

wherein

first all components are mixed with water, to obtain an emulsion having a water
phase and an oil phase,

then said emulsion is heated,

after the reaction temperature has been reached, a catalyst is added to the oil
phase of said emulsion, and

wherein

a) is at least one polyisocyanate,

b1) is at least one polyol comprising a structural unit $[-CH_2-CH_2-O-]_w-$, wherein w
is a positive integer from 10 to 200, wherein said structural unit $[-CH_2-CH_2-O-]_w-$ is obtained
from a synthesis component selected from the group consisting of ethylene glycol,
polyethylene glycol having a number average molecular weight of between 106 and 2000, and
ethylene oxide;

b2) is at least one polyol other than b1),

b3) is at least one compound containing at least two isocyanate-reactive groups
selected from the group consisting of thiol groups and primary and secondary amino groups,

b4) is at least one monofunctional monomer having an isocyanate-reactive group, and

c) is at least one ionic or potentially ionic synthesis component,

wherein the component c) is represented by the general formula $RG-R^1-DG$,
wherein RG is at least one isocyanate reactive group,
 DG is at least one actively dispersing group, and
 R^1 is an aliphatic, cycloaliphatic or aromatic radical comprising 1 to 20 carbon atoms;
wherein
the fraction of the structural units $[-CH_2-CH_2-O-]$, calculated at 44 g/mol, in the
polyol b1) is from 10 to 90% by weight, and
the fraction of the structural units $[-CH_2-CH_2-O-]$, calculated at 44 g/mol, in the
sum of the components a) + b1) + b2) + b3) + b4) + c) is at least 3% by weight.

Claim 55: A process for preparing a primary dispersion, said process comprising:
reacting the following components a), b1), and optionally b2), optionally b3),
optionally b4) and optionally c) in the presence of water, thereby obtaining an aqueous primary
dispersion, which comprises at least one polyurethane;

wherein

first all components are mixed with water, to obtain an emulsion having a water
phase and an oil phase, a catalyst is added to the water phase of said emulsion,
then said emulsion is heated, and

wherein

a) is at least one polyisocyanate,

b1) is at least one polyol comprising a structural unit $[-CH_2-CH_2-O-]_w$, wherein w
is a positive integer from 10 to 200, wherein said structural unit $[-CH_2-CH_2-O-]_w$ is obtained
from a synthesis component selected from the group consisting of ethylene glycol,
polyethylene glycol having a number average molecular weight of between 106 and 2000, and
ethylene oxide;

b2) is at least one polyol other than b1),
b3) is at least one compound containing at least two isocyanate-reactive groups
selected from the group consisting of thiol groups and primary and secondary amino groups,
b4) is at least one monofunctional monomer having an isocyanate-reactive group, and
c) is at least one ionic or potentially ionic synthesis component,
wherein the component c) is represented by the general formula $RG-R^1-DG$,
wherein RG is at least one isocyanate reactive group,
DG is at least one actively dispersing group, and
 R^1 is an aliphatic, cycloaliphatic or aromatic radical comprising 1 to 20 carbon atoms;
wherein
the fraction of the structural units $-[-CH_2-CH_2-O]-$, calculated at 44 g/mol, in the
polyol b1) is from 10 to 90% by weight, and
the fraction of the structural units $-[-CH_2-CH_2-O]-$, calculated at 44 g/mol, in the
sum of the components a) + b1) + b2) + b3) + b4) + c) is at least 3% by weight.

Claim 56: The process according to claim 27, wherein the component c) is not present.

IX. EVIDENCE APPENDIX

Exhibit A. Translation of DIN 53240 as filed on March 13, 2008.

Exhibit A

Catalysed acylation and titrimetric method of determining hydroxyl value

DIN
53240-2

ICS 71.040.40

Descriptors: Hydroxyl value, testing, paints, varnishes, resins, fats.

Bestimmung der Hydroxylzahl – Teil 2: Verfahren mit Katalysator

In keeping with current practice in standards published by the International Organization for Standardization (ISO), a comma has been used throughout as the decimal marker.

Foreword

This standard has been prepared by Technical Committee *Lackrohstoffe* of the *Normenausschuss Materialprüfung* (Materials Testing Committee) and the method it describes conforms in substance to method C-V 17a, 1989 edition, issued by the *Deutsche Gesellschaft für Fettwissenschaft* (German Association for the Science of Fats).

The symbol % is used in this standard to mean percentage by mass.

Unless otherwise specified, the reagents used shall be of analytical grade. For analytical procedures, the water used shall be distilled water or water of equivalent purity, and reagent solutions for which the solvent is not specified shall be solutions in such water.

1 Scope

The method described in this standard serves to determine the hydroxyl value of resins, paint and varnish raw materials, primary alcohols, glycols and fats. Its applicability to hydroxycarboxylic acids, phenolic hydroxyl groups, some polyols, such as trimethylolpropane, and substances containing aromatic groups which have been activated for Friedel-Crafts acylation shall be decided on a case-by-case basis.

2 Normative references

This standard incorporates, by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text, and the titles of the publications are listed below. For dated references, subsequent amendments to or revisions of any of these publications apply to this standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

- | | |
|-------------|---|
| DIN 8120-1 | Terminology associated with weighing machines – Classification – Scales and balances |
| DIN 12387 | Conical flasks for laboratory use |
| DIN 12664-1 | One-mark volumetric flasks with flanged rim, conical socket and conical joint, for laboratory use |
| DIN 12680-1 | Graduated cylinders for laboratory use |
| DIN 12700-1 | Burettes for laboratory use – General requirements |

Continued on pages 2 to 4.

Translation by DIN-Sprachendienst.

In case of doubt, the German-language original should be consulted as the authoritative text.

| | |
|---|---|
| DIN 53240 | Determination of hydroxyl number |
| DIN V 53242-1 | Sampling of raw materials for paints and varnishes – Concepts and general information |
| DIN 53242-2 | Sampling of liquid raw materials for paints and varnishes |
| DIN EN ISO 3682 | Binders for paints and varnishes – Determination of acid value – Titrimetric method (ISO 3682 : 1996) |
| ISO 5725-1 : 1994 | Accuracy (trueness and precision) of measurement methods and results – Part 1: General principles and definitions |
| ISO 5725-2 : 1994 | Accuracy (trueness and precision) of measurement methods and results – Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method |
| ISO 5725-3 : 1994 | Accuracy (trueness and precision) of measurement methods and results – Part 3: Intermediate measures of the precision of a standard measurement method |
| ISO 5725-4 : 1994 | Accuracy (trueness and precision) of measurement methods and results – Part 4: Basic methods for the determination of the trueness of a standard measurement method |
| ISO 5725-5 : 1998 | Accuracy (trueness and precision) of measurement methods and results – Part 5: Alternative methods for the determination of the precision of a standard measurement method |
| ISO 5725-6 : 1994 | Accuracy (trueness and precision) of measurement methods and results – Part 6: Use in practice of accuracy values |
| DGF Standard method C-V 17a (1989) <i>Hydroxylzahl (Bestimmung mit Essigsäureanhydrid)</i> (Hydroxyl value (determination with acetic anhydride)) | |

3 Concept

Hydroxyl value

The number of milligrams of potassium hydroxide, KOH, equivalent to the hydroxyl groups in 1 g of substance capable of acetylation with acetic acid.

4 Sampling

A composite sample shall be collected from the product under test as specified in DIN V 53242-1 and DIN 53242-2.

5 Number of tests

The hydroxyl value shall be determined at least twice.

6 Equipment

The following equipment shall be used.

6.1 Analytical balance (see DIN 8120-1).

6.2 250 ml conical flask with ground joint (e.g. DIN 12387-E 250 ANS 45 flask).

6.3 1 l volumetric flask (e.g. DIN 12664-1 volumetric flask).

6.4 250 ml measuring cylinder (e.g. DIN 12680-1 measuring cylinder).

6.5 25 ml pipette or dispenser.

6.6 10 ml burette, with 0,02 ml scale intervals (e.g. DIN 12700-1 burette) (alternatively, a suitable mechanized piston burette may be used).

6.7 50 ml burette, with 0,1 ml scale intervals (e.g. DIN 12700-1 burette) (alternatively, a suitable mechanized piston burette may be used).

In the case of potentiometric titration, the following shall be used.

6.8 Automatic titrator.

6.9 50 ml motorized piston burette.

6.10 Reference electrode/glass electrode system.

7 Reagents

The following reagents shall be used.

- 7.1 Acetic anhydride** (CAS No.*) 108-24-7).
- 7.2 Tetrahydrofuran** (CAS No. 109-99-9), stabilized with 2,6-di(*tert*-butyl)-4-methylphenol.
- 7.3 Dichloromethane** (CAS No. 75-09-2).
- 7.4 N,N-dimethylformamide** (CAS No. 68-12-2).
- 7.5 4-(dimethylamino)pyridine** (CAS No. 1122-58-3).
- 7.6 0,1 % phenolphthalein** in ethanol (95 % by volume) or **0,1 % thymolphthalein** in N,N-dimethylformamide.
- 7.7 Demineralized water.**
- 7.8 0,5 mol/l potassium hydroxide solution** in ethanol or methanol, for hydroxyl values not less than 5 mg KOH/g.
- 7.9 0,1 mol/l potassium hydroxide solution** in ethanol or methanol, for hydroxyl values less than 5 mg KOH/g.
- 7.10 Acetylating reagent**, prepared by introducing 500 ml of N,N-dimethylformamide into a 1 l volumetric flask, adding 125 ml of acetic anhydride and making up to the mark with N,N-dimethylformamide. This solution is colourless and will remain stable for one week if kept in a dark glass bottle.
- 7.11 Catalyst solution**, prepared by dissolving 25 g of 4-(dimethylamino)pyridine in 2,5 l of N,N-dimethylformamide at ambient temperature. This solution is colourless to yellowish (depending on the 4-(dimethylamino)pyridine used) and will remain stable for several months in a dark glass bottle.
- 7.12 Hydrolysing reagent**, prepared by mixing four parts by volume of N,N-dimethylformamide and one part by volume of demineralized water.

8 Procedure

The initial sample mass required for the analysis will depend on the anticipated hydroxyl value (see table 1 for guideline values), but shall be chosen so that about five moles of acetic anhydride are required for one hydroxyl equivalent.

Table 1: Initial sample mass (guideline values)

| Anticipated hydroxyl value, in mg KOH/g | | | | Initial sample mass, in g |
|--|-------|-------|-------|------------------------------|
| | | Up to | 15 | 10 |
| Over | 15 | up to | 20 | 6 |
| Over | 20 | up to | 25 | 5 |
| Over | 25 | up to | 30 | 4 |
| Over | 30 | up to | 40 | 3 |
| Over | 40 | up to | 50 | 2,5 |
| Over | 50 | up to | 100 | 2 |
| Over | 100 | up to | 200 | 1,2 |
| Over | 200 | up to | 300 | 0,6 |
| Over | 300 | up to | 400 | 0,4 |
| Over | 400 | up to | 500 | 0,3 |
| Over | 500 | up to | 750 | 0,2 |
| Over | 750 | up to | 1 000 | 0,15 |
| Over | 1 000 | | | 0,1 |

Weigh a test portion, to an accuracy of 0,1 %, into a conical flask and dissolve it in about 20 ml of tetrahydrofuran (or dichloromethane if it is not readily soluble in tetrahydrofuran). Pipette 25 ml of catalyst solution and then 10 ml of acetylating reagent, to an accuracy of 0,01 ml, into the conical flask and stopper it. Swirl the flask until its contents are homogeneous and allow it to stand undisturbed for at least 20 minutes at ambient temperature. The acetylation time will depend on the steric accessibility of the hydroxyl groups. If no empirical values are available for the material under test, check whether an acetylation time of 20 minutes is adequate. It should be noted, however, that a longer acetylation time may not result in a substantially increased hydroxyl value.

After acetylation, add 20 ml of hydrolysing reagent and shake the flask. Then allow the solution to stand for 30 minutes, swirling the flask occasionally.

*) Chemical Abstracts Service Registry Number.

Add 50 ml of tetrahydrofuran (or 50 ml of dichloromethane if this is used as the solvent) and ten drops of phenolphthalein or thymolphthalein solution and titrate the excess acid with alcoholic potassium hydroxide solution until the indicator colour change persists.

NOTE: For low hydroxyl values (below 10) or if the sample solutions are discoloured, titrate potentiometrically using a titrator which automatically titrates to the end point.

Determine the blank value by carrying out the same procedure under identical conditions in the absence of the test substance.

Determine the acid value separately by the method specified in DIN EN ISO 3682.

9 Evaluation

Calculate the hydroxyl value, *HV*, in mg KOH/g, using the following equation:

$$HV = \frac{(b - a) \cdot F}{E} + AV$$

where

a is the volume of potassium hydroxide solution required to titrate the sample, in ml;

b is the volume of potassium hydroxide solution required for the blank test, in ml;

E is the initial sample mass, in g;

F is the factor for converting millilitres of potassium hydroxide solution into milligrammes of potassium hydroxide (28,05 for a 0,5 mol/l KOH solution and 5,61 for a 0,1 mol/l KOH solution);

AV is the acid value, in mg KOH/g.

10 Precision

10.1 General

An interlaboratory test carried out in March 1990 by the responsible technical committee and based on the standards of the ISO 5725 series yielded the values for the repeatability and reproducibility limits given below. However, no samples having hydroxyl values greater than 200 mg KOH/g were tested.

10.2 Repeatability limit

Two successive analyses performed on the same day by the same operator using the same equipment on different initial sample masses from the same sample material shall differ by no more than 0,84 mg KOH/g for hydroxyl values below 20 and by no more than 1,72 mg KOH/g for hydroxyl values between 20 and 200.

10.3 Reproducibility limit

Two analyses performed in different laboratories on the same sample material shall differ by no more than 1,36 mg KOH/g for hydroxyl values below 20 and by no more than 3,28 mg KOH/g for hydroxyl values between 20 and 200.

11 Test report

The test report shall refer to this standard and include the following details:

- nature and description of product tested;
- hydroxyl value, in mg KOH/g, stating the method of titration (potentiometric or using an indicator);
- acetylation time;
- test results (single values and mean);
- any deviation from the specified method;
- date of testing.

Bibliography

List of some standardized and non-standardized methods of determining hydroxyl values

| | |
|-----------------|---|
| DIN 53240 | Determination of hydroxyl value |
| DIN EN 1240 | Adhesives – Determination of hydroxyl value and/or hydroxyl content |
| ISO 2554 : 1997 | Plastics – Unsaturated polyester resins – Determination of hydroxyl value |
| ISO 4327 : 1979 | Non-ionic surface active agents – Polyalkoxylated derivatives – Determination of hydroxyl value – Phthalic anhydride method |
| ISO 4629 : 1996 | Binders for paints and varnishes – Determination of hydroxyl value – Titrimetric method |
| ISO 6796 : 1981 | Polyglycols for industrial use – Determination of hydroxyl value – Phthalic anhydride esterification method |
| ISO 7142 : 1984 | Binders for paints and varnishes – Epoxy resins – General methods of test |

X. RELATED PROCEEDINGS APPENDIX

None.